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MELLON INSTITUTE

Report No. 13

MONTHLY SCIENTIFIC PROGRESS REPORT

For the month of July 1961

STRESS CORROSION OF HIGH STRENGTH STEELS AND ALLOYS; ARTIFICIAL ENVIRONMENT

Research Project No. 389-I

Sponsored by
U. S. Army Ordnance, Frankford Arsenal
Mr. H. Rosenthal, Contract Monitor

Contract No. DA-36-034-ORD-3277RD

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I. INTRODUCTION

The project work described herein represents a portion of a grant made available by the Army to promote a general scientific advancement in the area of case materials for missiles. This specific project is concerned with the synthetic environment stress corrosion testing of specified high strength steels and alloys. The research objectives of the project were presented in the July Monthly Scientific Report, Report No. 1.

Natural environmental tests on high strength steels and alloys are being conducted by Aerojet General Corporation, with actual production environments and rocket propellant environments being utilized. By prior mutual agreement, the same steel and alloy sheet material will be used for both projects and possible heat treatment variations will be circumvented by exchanging heat treated material whenever possible.

A number of drawings and schematic diagrams of apparatus and test methods pertinent to the project have been presented in prior reports. In addition, summarized surveys of applicable industrial and military literature have also been presented previously (August and September re[illegible]

This report presents further information on continuing U-bend and bent beam tests for the assigned alloys, and for Rocoloy 270, 4137 Co, and Ardeform 301 missile steels.

II. EXPERIMENTAL PROCEDURES

Test Methods

A discussion of the U-bend and bent beam test methods together with an outline of the synthetic stress corrosion test environments used in performing the research are given in the July, 1960, Monthly Scientific Report, Report No. 1.

All recently-exposed (and future) samples being subjected to stress corrosion testing are being weighed prior to exposure to the test solutions to provide, whenever possible, general corrosion information.

Apparatus

Schematic drawings of bent beam sample holders, the U-bend test and holders, a sample bending device for bent beam specimens, and a stress corrosion test tank were presented in Report Nos. 1 and 3.

Polyethylene containers are presently being used for the stress corrosion exposure of U-bend specimens to the various synthetic environments. Each container will adequately hold six U-bend specimens. The use of these containers will supplement the samples presently being tested in epoxy-coated tanks.

Construction of shelving and an aeration system (Report No. 6) together with other pertinent items has facilitated (wherever

feasible) the transfer of samples under test from the glass containers to the epoxy-coated test tanks. The completed facility is illustrated in Report No. 7.

Alloy Sample Material

The alloys being used in the study on stress corrosion include:

1. Low Alloy: Ladish D6Ac.
2. Si-Modified 4300 Series: 300 M.
3. Hot-Worked Die Steel: Vascojet 1000.
4. Cold-Worked PH Steel: AM 355.
5. Heat-Treated PH Steel: PH15-7 Mo.
6. Titanium Alloy: B120VCA.
7. Low Alloy-Cobalt Modified: 4137 Co.
8. Low Alloy-Cobalt Modified: Rocoloy 270.
9. Stretch-Formed 17/7 Stainless Steel: Ardeform 301.

All of the sample material is being tested in sheet form and was procured as such.

Chemical analyses for the above listed alloys and for the comparative heats are given in their entirety in Table I. Physical properties and heat treating surveys for the alloys were presented in Report No. 12.

The status of the alloy sample material is as follows:

1. D6Ac - ready for final machining
2. 300M - ready for final machining
3. Vascojet 1000 - ready for heat treatment
4. AM355 - under test
5. PH15-7Mo - ready for heat treatment
6. B120VCA - being machined
7. 4137 Co - under test
8. Rocoloy 270 - ready for final machining
9. Ardeform 301 - under test

On all of the above listed alloys, the final machining step consists of wet grinding the specimens to thickness (.051 ± 001). After heat treating, all specimen surfaces (except ends) are hand polished with 240-grit emery paper.

III. EXPERIMENTAL

Sample preparation on all alloys noted in the aforementioned section is continuing.

U-bend stress corrosion testing is continuing for the alloys, including 4137 Co, AM355, PH15-7Mo, B120VCA and Ardeform 301. Bent beam specimens of 4137 Co and AM355 are still under test.

The foregoing specimens under test are discussed in greater detail as follows:

Bent Beam Tests

Bent beam specimens of AM355 (secondary direction) are undergoing stress corrosion testing. These specimens were cold-rolled to their strength level (250, 261, 302 Kpsi) by the vendor. To date there have been no failures, as indicated in Table II. Visual observation shows no indication of general corrosion on the AM355 specimens. It is expected in the near future that the primary direction of the AM355 bent beam specimens will also be under test.

In the cooperative program with U. S. Steel Applied Research Laboratory, in which bent beam specimens of 4137 Co specimens were exposed to three natural environments, little change has been noted in the status of the specimens.

The U. S. Steel Applied Research program utilizes samples of 4137 Co heat treated to three strength levels and then stressed to 75% of their Y.S. The specimens were exposed to two natural environments: Kure Beach, N.C. (marine) and Monroeville (semi-industrial). Table III gives the cumulative data on the specimens to date. It may be noted that the status of the specimens has not changed over the past few months.

The program on 4137 Co bent beam specimens conducted by this Project utilized the same heat of sample material and the same heat-treating procedures. An industrial environment, Pittsburgh, Pa., was chosen for testing. Specimens of the three Y.S. levels were stressed to 50%, 75% and 90% of their Y.S. The cumulative results on the specimens used by project personnel is given in Table IV. It may be noted that very little change has taken place over the last few months and only scattered failures have been reported.

U-Bend Tests

The U-bend specimens of the high Y.S., primary direction, of AM355 are presented in Table V. Since the specimens are only from one strength level and one direction, no correlation can be made between U-bend and bent beam results for AM355. The remainder of the U-bends for both directions and all strength levels are expected to be under test in the near future.

It can be seen from Table VI that no failures have occurred with the B120VCA U-bends. These specimens are, however, from the lowest strength level (135 Kpsi) and the secondary direction.

The U-bend specimens of the PH15-7Mo alloy are still undergoing testing. It is shown in Table VII that no failures have occurred to date. The specimens were taken in both the primary and secondary directions and were cold-rolled to a strength level of approximately 195 Kpsi by the vendor.

Additional U-bend specimens of PH15-7Mo and AM355 are being tested in three additional test solutions above and beyond those assigned for this project. These test solutions include:

1. a chlorinated non-hydrocarbon (CCl_4);
2. a non-chlorinated hydrocarbon (C_5H_{12}); and
3. a chlorinated hydrocarbon ($C_2H_2Cl_4$).

Table VIII gives the cumulative results on the aforementioned test. No recent failures have resulted from the foregoing tests, exclusive of the one early failure of a PH15-7Mo sample in CCl_4.

Ardeform 301 specimen testing is continuing in the five test solutions, namely: NaCl, $NaNO_3$, Na_2SO_4, $NaPO_3$ and Na_2S. The cumulative data on sample No. 1 of the Ardeform 301 material is given in Table IX. The results indicate a susceptibility of the Ardeform 301 in the one molar NaCl test solution. Sample No. 2 of the Ardeform 301

does not show the same degree of susceptibility as shown in Table X. Monthly Progress Report No. 10 presents the sample preparation information for Samples 1 and 2.

U-bend tests on 4137 Co, a low alloy missile steel, are continuing. It can be seen from Table XI that the specimens in the one molar $NaNO_3$ and $NaPO_3$ have failed for all three strength levels. The Na_2S test solution appears to have only a slight effect on the 4137 Co specimens.

The 4137 Co results on the effect of water vapor exposure are presented in Table XII. As the data shows, the "Humid Air" test was the only one with an effect on the specimens. There have been no failures with the specimens tempered at 1100°F in the "Humid Air."

IV. FUTURE WORK

Sample preparation, heat treatment, metallographic examination, and stress corrosion testing will constitute the main effort of the project.

Photo-micrographs of specimens from the heat treatment survey will be presented together with grain size determinations.

Cumulative test results on bent beam and U-bend tests will be presented as they become available.

Future reports will be presented on a quarterly rather than a monthly basis.

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TABLE I

CHEMICAL ANALYSES OF STEELS AND ALLOYS

Steel or Alloy	Sample Designation	Chemical Composition, % by wt.														
		C	Mn	P	S	Si	Cr	Ni	Mo	Al	V	Co	W	N	Ti	Fe
D6Ac	Primary Heat	0.48	0.68	0.007	0.008	0.22	1.23	0.58	1.01		0.06					bal.
D6Ac	Comp. Ht #2	0.48	0.70	0.010	0.002	0.19	1.19	0.62	1.08		0.10					bal.
300M	Primary Heat	0.43	0.66	0.006	0.005	1.80	0.84	1.83	0.35	0.065	0.06					bal.
300M	Comp. Ht #1	0.41	0.70	0.007	0.004	1.50	0.77	1.71	0.37	0.11	0.09					bal.
V-1000	Primary Heat	0.38	0.28	0.010	0.008	0.80	5.10		1.35		0.48					bal.
V-1000	Comp. Ht #1	0.38	0.22	0.009	0.007	0.90	5.19		1.20		0.50					bal.
V-1000	Comp. Ht #2	0.39	0.27	0.009	0.008	0.98	5.13		1.29		0.51					bal.
V-1000	Comp. Ht #3	0.42	0.36	0.011	0.006	0.96	4.90		1.28		0.50					bal.
AM355	Primary Heat	0.14	0.72	0.018	0.018	0.29	15.60	4.38	2.71					0.11		bal.
PH15-7Mo	Primary Heat	0.08	0.54	0.014	0.008	0.26	15.05	7.12	2.16	1.16						bal.
B120VCA	Primary Heat	0.02					11.2	0.02		3.0	13.7		0.018		bal.	0.23
B120VCA	Comp. Ht #1	0.03					10.01	0.07		2.95	13.30		0.012		bal.	0.68
4137Co	Vac. Melt	0.42	0.77	0.009	0.008	0.90	1.14	0.12	0.26	0.02	0.15	7.10				bal.
R270	Air Melt	0.48	1.01	0.008	0.007	1.38	0.95	1.18	0.45	0.12	0.26	0.96				bal.
R270	Vac. Melt	0.46	0.96	0.009	0.006	1.08	1.65	1.29	0.54	0.038	0.20	1.02				bal.
Ardo. 301	Cold Wkd S.S.	0.06	1.52	0.030	0.016	0.49	17.02	7.50								bal.

TABLE II

CUMULATIVE AM355 STRESS CORROSION BENT BEAM TESTS

Secondary (Highest Y.S.) Direction*

Test Solution	Approx. Tensile Stress on Sample**	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
NaCl, 1 M	180 kpsi	6	none to 153 days	--	--
	195	6	none to 137 days	--	--
	226	6	none to 150 days	--	--
Na_2SO_4, 1 M	180	6	none to 153 days	--	--
	195	6	none to 137 days	--	--
	225	6	none to 150 days	--	--
$NaNO_3$, 1 M	180	6	none to 153 days	--	--
	195	6	none to 137 days	--	--
	225	6	none to 150 days	--	--
$NaPO_3$, 1 M	180	6	none to 153 days	--	--
	195	6	none to 137 days	--	--
	225	6	none to 150 days	--	--
Na_2S, 1 M	180	6	none to 153 days	--	--
	195	6	none to 137 days	--	--
	225	6	none to 150 days	--	--

* All samples stressed in holder to 75% of Y.S.

** The three Y.S. test levels of as-rolled material: 250 kpsi, 261 kpsi; and 302 kpsi.

TABLE III

CUMULATIVE 4137 Co STRESS CORROSION BENT BEAM TESTS*

Primary (Lowest Y.S.) Direction

Austenitizing Temperature	Tempering Temperature	Approximate Tensile Stress**	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
Marine Exposure (Kure Beach, North Carolina)						
1700°F	550°F	190 kpsi	5	3	8	3 to 16
	750	160	5	5	13	6 to 18
	1100	130	5	None to date	--	--
tests begun 10-26-60						
Semi-Industrial Exposure (Monroeville, Pennsylvania)						
1700°F	550°F	190 kpsi	5	5	19	2 to 30
	750	160	5	5	35	18 to 45
	1100	130	5	None to date	--	--
tests begun 10-26-60						

* Cooperative testing program with U. S. Steel Applied Research Lab., Monroeville, Pa.
** All samples surface-stressed in holder to 75% of Y.S.

TABLE IV

CUMULATIVE 4137 Cr STRESS CORROSION BENT BEAM TESTS

Primary (Lowest Y.S.) Direction

Austenitizing Temperature	Tempering Temperature	% of Y.S. for Test	Approximate Tensile Stress	Number of Samples	Failures to Date	Average Time to Failures, days	Failure Time Range, days
Severe Industrial Exposure (Pittsburgh, Pennsylvania)							
1700°F	550°F	50	130 kpsi	6	1 to 151 days	--	--
	750	50	115	6	none to 151 days	--	--
	1100	50	95	6	none to 151 days	--	--
1700	550	75	195	6	6	47.2	30 to 65
	750	75	175	6	1 to 151 days	--	--
	1100	75	140	6	none to 151 days	--	--
1700	550	90	230	6	6	33.6	21 to 43
	750	90	200	6	3 to 151 days	--	--
	1100	90	170	6	none to 151 days	--	--

tests begun 3-2-61

TABLE V

CUMULATIVE AM355 STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Test Solution	Y.S. (.2% Offset) Test Level, Kpsi	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
NaCl	226 (as recvd)	6	6	3.25	0.5 to 8
$NaNO_3$	226 (as recvd)	6	none to 118 days	--	--
Na_2S	226 (as recvd)	6	none to 116 days	--	--
Na_2SO_4	226 (as recvd)	6	none to 118 days	--	--
$NaPO_3$	226 (as recvd)	6	none to 116 days	--	--

Note: Outer surface of U-bend specimens stressed beyond the Y.S.

TABLE VI

CUMULATIVE 5120 VCA STRESS CORROSION U-BEND TESTS

Secondary (Highest Y.S.) Direction

Test Solution	Y.S. (.2% Offset) Test Level, Kpsi	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range (days)
NaCl, 1 M	135 (as recvd)	4	none to 207 days	--	--
$NaNO_3$, 1 M	135 (as recvd)	4	none to 207 days	--	--
$NaPO_3$, 1 M	135 (as recvd)	4	none to 207 days	--	--

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

TABLE VII

CUMULATIVE PH15-7 Mo STRESS CORROSION U-BEND TESTS

Test Solution	Direction of Specimen	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range (days)
NaCl	Primary	6	none to 124 days	--	--
	Secondary	6	none to 124 days	--	--
$NaNO_3$	Primary	5	none to 124 days	--	--
	Secondary	6	none to 124 days	--	--
Na_2S	Primary	6	none to 124 days	--	--
	Secondary	6	none to 124 days	--	--
Na_2SO_4	Primary	5	none to 124 days	--	--
	Secondary	6	none to 124 days	--	--
$NaPO_3$	Primary	5	none to 124 days	--	--
	Secondary	6	none to 124 days	--	--

Note: Outer surface of U-bend specimens stressed beyond the Y.S.
Primary (lowest Y.S.) and Secondary (highest Y.S.).

TABLE VIII

CUMULATIVE PH15-7 Mo AND AM355 STRESS CORROSION U-BEND TESTS

Stress Corrosion Environment	Type of Steel	Sample Direction	Y.S. Kpsi	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, days
Carbon Tetrachloride CCl_4 (Chlorinated nc.: hydrocarbon)	PH15-7Mo	Primary	201	6	1 to 52 days	--	--
	PH15-7Mo	Secondary	218	6	none to 52 days	--	--
	AM355	Primary	226	6	none to 52 days	--	--
	AM355	Primary	215	6	none to 52 days	--	--
Pentane C_5H_{12} (Non chlorinated hydrocarbon)	PH15-7Mo	Primary	201	6	none to 52 days	--	--
	PH15-7Mo	Secondary	218	6	none to 52 days	--	--
	AM355	Primary	226	6	none to 52 days	--	--
	AM355	Primary	215	6	none to 52 days	--	--
Trichlorethane C_2H_3Cl (Chlorinated hydrocarbon)	PH15-7Mo	Primary	201	5	none to 52 days	--	--
	PH15-7Mo	Secondary	218	6	none to 52 days	--	--
	AM355	Primary	226	6	none to 52 days	--	--
	AM355	Primary	215	6	none to 52 days	--	--

Note: Outer surface of U-bend specimens stressed beyond the Y.S.

TABLE IX

CUMULATIVE ARDEFORM 301 STRESS CORROSION U-BEND TESTS

Stress Corrosion Environment	Sample Direction	Test Surface in Tension	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
Sample No. 1						
NaCl, 1 M	Longitudinal	Outside (convex)	4	4	1.25	0.5 to 2
	Longitudinal	Inside (concave)	4	2 to 118 days	--	--
	Trans.	Outside	6	none to 118 days	--	--
$NaNO_3$, 1 M	Long.	Outside	4	none to 118 days	--	--
	Long.	Inside	4	none to 118 days	--	--
	Trans.	Outside	6	none to 118 days	--	--
Na_2S, 1 M	Long.	Outside	4	none to 118 days	--	--
	Long.	Inside	4	none to 118 days	--	--
	Trans.	Outside	6	none to 118 days	--	--
Na_2SO_4, 1 M	Long.	Outside	4	none to 118 days	--	--
	Long.	Inside	4	none to 118 days	--	--
	Trans.	Outside	6	none to 118 days	--	--
$NaPO_3$, 1 M	Long.	Outside	4	none to 118 days	--	--
	Long.	Inside	4	none to 118 days	--	--
	Trans.	Outside	6	none to 118 days	--	--

Note: Outer surface of U-bend samples is stressed beyond Y.S.

TABLE X

CUMULATIVE ARDEFORM 301 STRESS CORROSION U-BEND TESTS

Stress Corrosion Environment	Sample Direction	Test Surface in Tension	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
Sample No. 4						
NaCl, 1 M	Longitudinal	Outside (convex)	6	none to 61 days	--	--
	Longitudinal	Inside (concave)	4	1 to 61 days	--	--
	Transverse	Outside	5	none to 61 days	--	--
$NaNO_3$, 1 M	Long.	Outside	5	none to 66 days	-	--
	Long.	Inside	4	none to 66 days	--	--
	Trans.	Outside	5	none to 66 days	--	--
Na_2S, 1 M	Long.	Outside	4	none to 66 days	--	--
	Long.	Inside	-	---	--	--
	Trans.	Outside	5	none to 66 days	--	--
Na_2SO_4, 1 M	Long.	Outside	4	none to 66 days	--	--
	Long.	Inside	3	none to 66 days	--	--
	Trans.	Outside	5	none to 66 days	--	--
$NaPO_3$	Long.	Outside	4	none to 66 days	--	--
	Long.	Inside	4	none to 66 days	--	--
	Trans.	Outside	5	none to 66 days	--	--

Note: Outer surface of U-bend samples is stressed beyond Y.S.

TABLE XI

CUMULATIVE 4137 Co STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Test Solution	Austenitizing Temperature	Tempering Temperature	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
NaCl, 1 M	1700°F	550	6	6	1.3	0.5 to 1.5
		750	6	4 to 375 days	--	--
		1100	6	1 to 375 days	--	--
Na_2SO_4, 1 M	1700°F	550	6	6	0.[illegible]	10 min. to 2.5 days
		750	6	none to 315 days	--	--
		1100	6	1 to 315 days	--	--
$NaNO_3$, 1 M	1700°F	550	6	6	1.2	0.5 to 1.5
		750	6	6	33.8	29.5 to 39.5
		1100	6	6	120.1	26.0 to 277.0
$NaPO_3$, 1 M	1700°F	550	6	6	4.5 min.	3 to 6 min.
		750	6	6	0.3 days*	34 min. to 5 days
		1100	6	6	1.5 days**	0.5 to 27 days
Na_2S, 1 M	1700°F	550	6	2 to 181 days	--	--
		750	6	1 to 181 days	--	--
		1100	6	none to 180 days	--	--

* One specimen lasting 5 days not averaged.
** One specimen lasting 27 days not averaged.

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

TABLE XII

CUMULATIVE 413T Co STRESS CORROSION U-BEND TESTS

Primary (Lowest Y.S.) Direction

Environment	Austenitizing Temperature	Tempering Temperature	Number of Samples	Failures to Date	Average Time to Failure, days	Failure Time Range, (days)
Dry Air (dessicator)	1700°F	550	3	none to 181 days	--	--
		750	3	none to 181 days	--	--
		1100	3	none to 181 days	--	--
Humid Air (satur. with water vapor)	1700°F	550	4	4	6.4	4 to 11.5
		750	4	4	24.8*	2.5 to 143
		1100	4	none to 173 days	--	--
Laboratory (exposed directly to lab. environ.)	1700°F	550	4	none to 173 days	--	--
		750	4	none to 173 days	--	--
		1100	4	none to 173 days	--	--

* One specimen lasting 143 days not averaged.

Note: Outer surface of U-bend samples is stressed beyond the Y.S.

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